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Fracture Toughness and Stress Corrosion Resistance of U-0.75 wt% Ti

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Introduction

Late in 1978, ARL-MD, Watertown was asked to participate in an investigation of several failures on launch of depleted-uranium cored XM774 rounds during low temperature firing. Failure occurred in the vicinity of the rear-most buttress groove of the core where the fillet stress approximates the yield strength of the U-0.75 wt% Ti core alloy. A simple fracture mechanics approach suggested that poor low temperature fracture toughness of the core alloy was contributory.

As a consequence, a systematic investigation of the fracture toughness of the currently produced U-0.75 wt% Ti core alloy was carried out. The U-0.75 wt% Ti alloy was provided by National Lead of Ohio (NLO) and Battelle Northwest (BNW). The failed cores were processed by NLO. The XM833 U-0.75 wt% Ti core material was also obtained from Rocky Flats (RF) for comparison. Representative cores from each source were fully characterized and processing parameters, mechanical properties, microstructure, and test temperature were correlated with fracture toughness.

Materials

The NLO XM774 penetrators were fabricated from a 1.4 in. diameter rod which was rolled from 8 in. diameter ingots. The bars were solution treated for 10 minutes at 899°C in NUSAL, plunge oil quenched, and aged at 350°C in a lead bath.

Six bars, 6 in. long and 1.4 in. in diameter, were received from BNW. These bars were the bottom portions of longer 16 in. bars and the first to enter the water on vertical quench. The 16 in. long extruded bars were vacuum solution treated at 800°C for two hours and 850°C for one-half hour, vertically water quenched at 18 in. per minute, and aged at 350°C in a lead bath for 16 hours.

The RF XM833 penetrators were fabricated from 1.4 in. diameter bars which were alpha extruded from 4 in. diameter ingots. The ingots were homogenized in vacuum at 1050°C for six hours prior to extrusion. The extruded bars were then solution treated for two hours at 800°C and one-half hour at 850°C, vertically water quenched at 18 in. per minute, and aged at 350°C in a lead bath for 16 hours.

Four additional 1.4 in. diameter bars which were received from NLO in the as-rolled condition were given STA treatments comparable to BNW and RF processing; i.e., they were vacuum solution treated at ARL-MD, Watertown for two hours at 800°C and one-half hour at 850°C, vertically quenched in water at 21 in. per minute, and aged in vacuum at 350°C, 370°C, and 390°C, respectively, for seven hours.

Fracture Toughness Test Procedures

Sampling

Two types of fracture toughness specimens were utilized: (1) a single edge-notched bend specimen conforming to plane strain requirements (K_{IC}) of ASTM E 399-74 (FT1); and (2) a slow-bend V-notched Charpy impact specimen (CV2) for approximate K_{IC} or K_Q . Both types of specimens were used for **static** fracture toughness measurements. Regardless of the type of specimen, the notches were always machined from the outer diameter of the bar or penetrator core so that the microstructure in the vicinity of the notch would be comparable to that of the penetrator buttress groove.

From each of four XM774 penetrators representative of NLO lots which failed on low temperature firing, two Charpy, K_Q specimens and two K_{IC} specimens were cut alternately starting at the nose of the penetrator; i.e., the end which entered the water first during the vertical quench. A total of four Charpy and four K_{IC} specimens were cut per penetrator. In a similar fashion, four K_{IC} specimens and four K_Q specimens were machined from three XM833 RF penetrators. Based on the similarity of K_{IC} and K_Q values obtained, it was decided to concentrate on the simplest and least costly specimen, the V-notch bend Charpy impact specimen only, and report K_Q values for the remaining materials evaluated. Therefore, only K_Q specimens were machined from four NLO as-rolled bars which had been vacuum solution treated, vertically water quenched, and aged at ARL-MD, Watertown and from six BNW bars which were similarly heat treated. In addition, tension and K_{ISCC} specimens were fabricated from the above materials to confirm the specified strength requirement and to determine susceptibility to stress corrosion cracking.

The stress corrosion specimens (see Figure 1) which were single edge notch specimens (3.0 x 0.20 x 0.20 in.) were cut with the long dimension parallel to the direction of maximum grain flow and notched so that crack growth and fracture would occur in the radial direction.

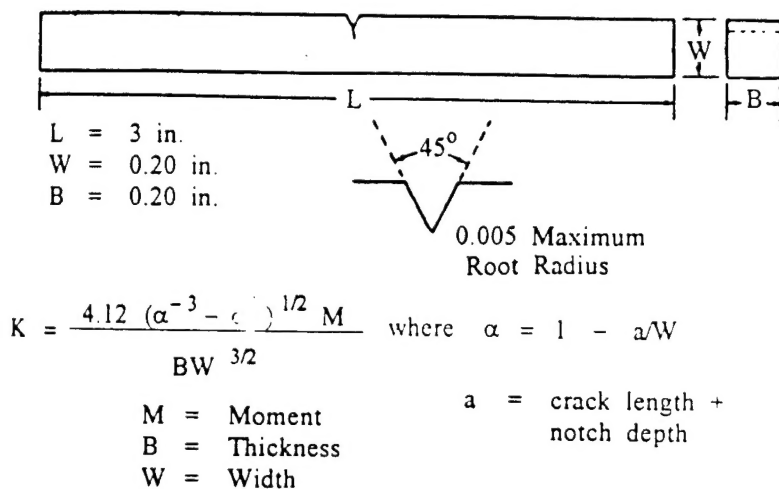


Figure 1. Specimen geometry and equation for K values.

Test Method

The procedure for KIC measurement involved three-point bend testing of notched specimens that had been precracked in fatigue. Load versus displacement across the notch was recorded autographically. The KIC value was calculated from the load corresponding to a 2% increment of crack extension by equations which have been established on the basis of elastic stress analysis of bend specimens. The detailed procedure is described in ASTM E 399-74. The method for KQ measurement employed a Charpy specimen provided with a sharp notch terminating in a fatigue crack tested in three-point bending. The maximum load in the test was recorded and the nominal crack strength was determined from this value as well as the original dimensions of the specimen using the single beam equation. A detailed description is contained in the proposed E24.03.03 draft dated February 7, 1979. Precracking of specimens for both test procedures involved initiation of the crack and subsequent growth in tension.

The method for stress corrosion measurements follows. The test uses a precracked bar stressed as a cantilever beam. A sharp notch is machined across the rectangular bar specimens at mid-length, and is sharpened by fatiguing. The specimen is held in a rack horizontally with the precracked central portion surrounded by a plastic bottle which contains the environment. One end of the specimen is clamped to the mast of the rack and the other end to

an arm from which weights are suspended. On evaluating the alloy, the specimen is first stressed in air at increasing loads until it fractures. The data are reduced to stress intensity using the Kies equation (see Figure 1). Having established stress intensity for dry conditions (K_{IC}), a specimen is similarly tested in distilled H_2O and NaCl solutions at a somewhat lower stress intensity. If the specimen did not fail within an hour, the stress intensity was increased by approximately 3% each succeeding hour until failure occurred and the time required for rupture noted. Additional specimens were stressed at decreasingly lower stress intensities for 1000 hours or until failure occurred to give a more valid value for K_{ISCC} , which was determined from a plot of stress intensity versus time to failure. K_{ISCC} is the threshold stress intensity value for the onset of cracking.

Results

Comparison of Failed NLO XM774 Penetrators Versus the RF XM833 Processed Material

Chemistry, Microstructure, Mechanical Properties

Table 1 summarizes mechanical properties and chemistries for NLO XM774 and RF XM833 penetrators. Major differences were observed in hydrogen content, elongation, and RA values. The NLO material exhibited higher H and lower elongation and RA.

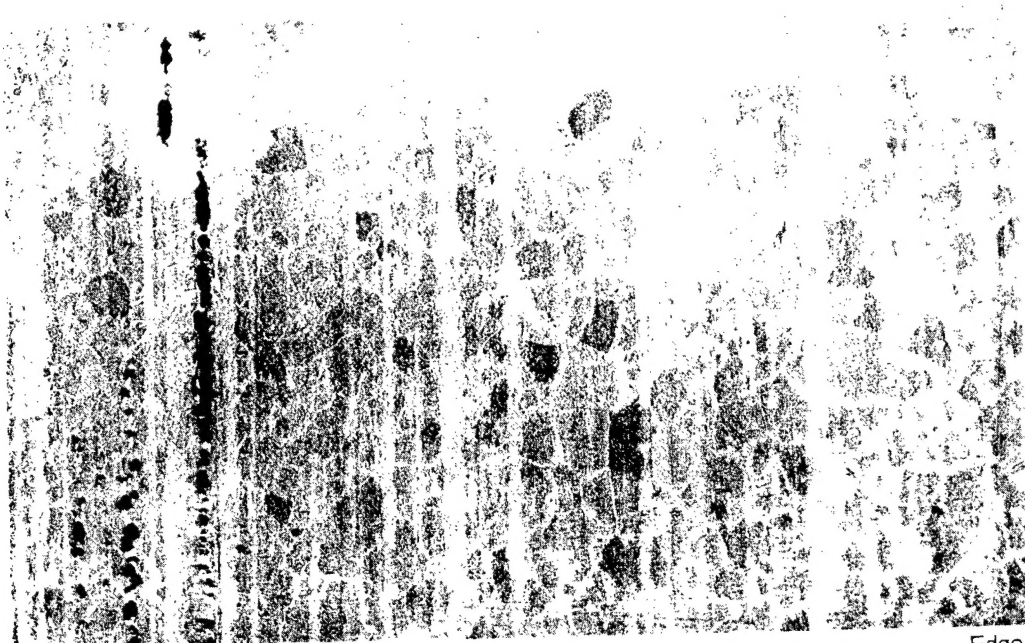
Table 1. XM774 Staballoy properties

	RF	NLO
Ultimate (ksi)	210	196
Yield (ksi)	115	114
Elongation (%)	12 - 16	5 - 9
RA (%)	12 - 16	4 - 8
Hardness (HR _C)	38 - 43	40 - 42
Ti (%)	0.69 - 0.73	0.69 - 0.71
C (ppm)	<100	<40
H (ppm)	<1	2 - 4

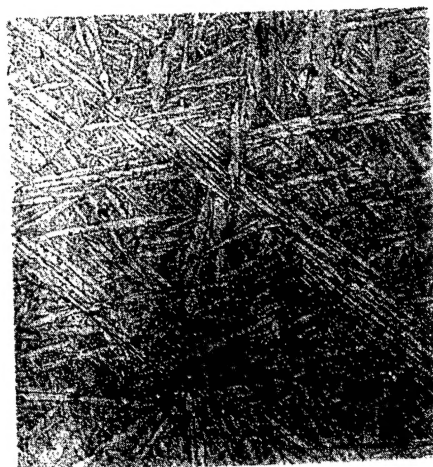
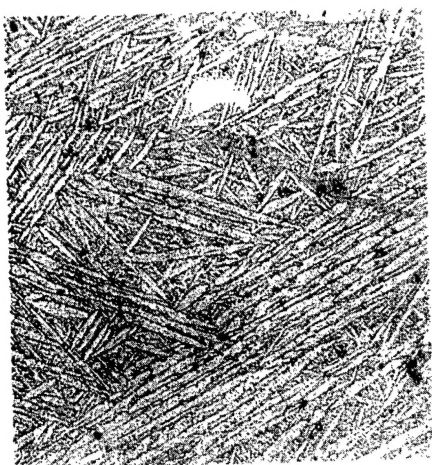
The structure of the NLO bars is shown in Figure 2. The view is perpendicular to the extrusion direction at the diameter and represents slightly more than one-half of the complete cross section. A coarse duplex grain size is observed along with banding and center-line porosity or voids.

The microstructure of an XM833 penetrator is shown at both the nose section from the bar entering the water first on vertical quenching

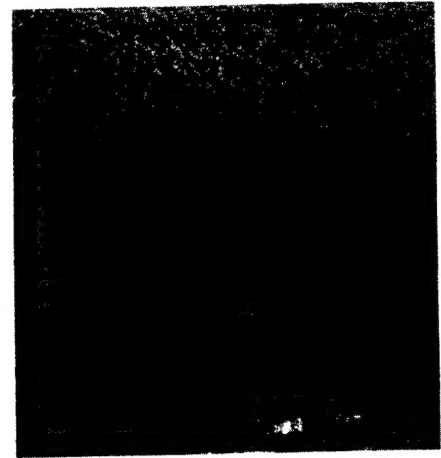
(see Figure 3) and at the tail, or rear, portion of the bar which entered the water last (see Figure 4). The microstructure in Figure 3 is essentially martensitic with evidence of incipient slack quench at the grain boundaries and small voids, particularly in the central area are observed. The tail or rear, views show a more pronounced slack quench and even larger voids, particularly in the central areas (see Figure 4).



Center Edge
Figure 2. U-0.75 wt% Ti (NLO) - solution treated (molten salt) 899°C for 10 minutes, oil quenched, and aged at 350°C for one hour. Mag. 9X.



Center Edge
Figure 3. U-0.75 wt% Ti (RF) nose section - solution treated at 800°C for two hours, 850°C for one-half hour; vertically water quenched 18 inches per minute; aged (lead bath) for 16 hours at 350°C. Mag. 100X



Center

Edge

Figure 4. U-0.75 wt% Ti (RF) tail section - solution treated at 800°C for two hours, 850°C for one-half hour, vertically water quenched 18 inches per minute; aged (lead bath) for 16 hours at 350°C. Mag. 100X

Fracture Toughness Versus Temperature

Figure 5 compares fracture toughness data for the failed NLO penetrator material obtained from the two types of specimens employed. The data was designated K_{IC} if all the conditions of ASTM E 399-74 were met; otherwise, the values were designated K_Q .

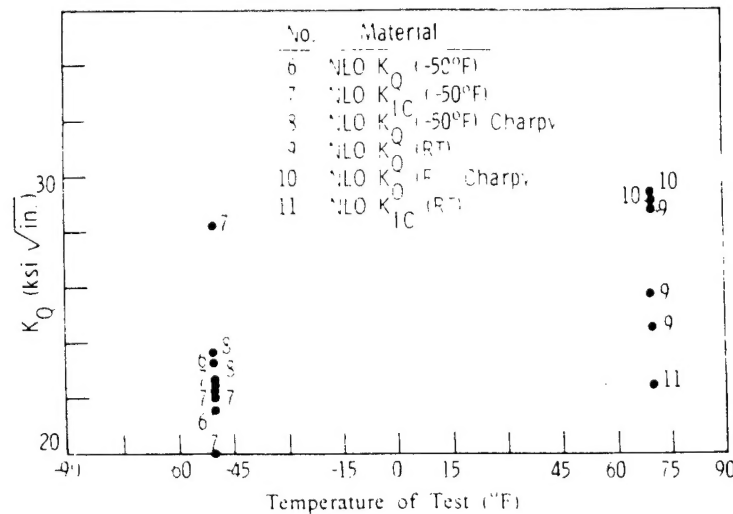


Figure 5. Fracture toughness of aged U-0.75 wt% Ti NLO penetrators versus temperature of test.

All K_{IC} and K_Q values were below 30 ksi√in. regardless of test temperature. The K_{IC} and K_Q values were in fair agreement. The average value at -50°F was 22 ksi√in., and at 75°F, 27 ksi√in.

Previous work at ARL-MD, Watertown has shown that fracture toughness values for titanium and steel alloys obtained with compact tension and bend specimens conforming to ASTM E 399-74, were in good agreement with those obtained with precracked Charpy specimens up to values of $40 \text{ ksi}\sqrt{\text{in.}}$ (2,3).

Fracture Toughness Versus Hardness

Figure 6 shows a plot of fracture toughness versus HRC hardness values for individual specimens taken from the NLO failed XM774 penetrator lots and the RF XM833 penetrators. The slightly softer vacuum solution treated, and vertically water quenched XM833 penetrators had significantly higher toughness values than the NLO XM774 penetrator lots which were molten salt solution treated, plunge quenched in oil, and had high hydrogen. At both room temperature and -50°F , fracture toughness values for specimens from RF XM833 penetrator lots were greater than $35 \text{ ksi}\sqrt{\text{in.}}$. All values were below $30 \text{ ksi}\sqrt{\text{in.}}$ for specimens from the NLO XM774 penetrator lots.

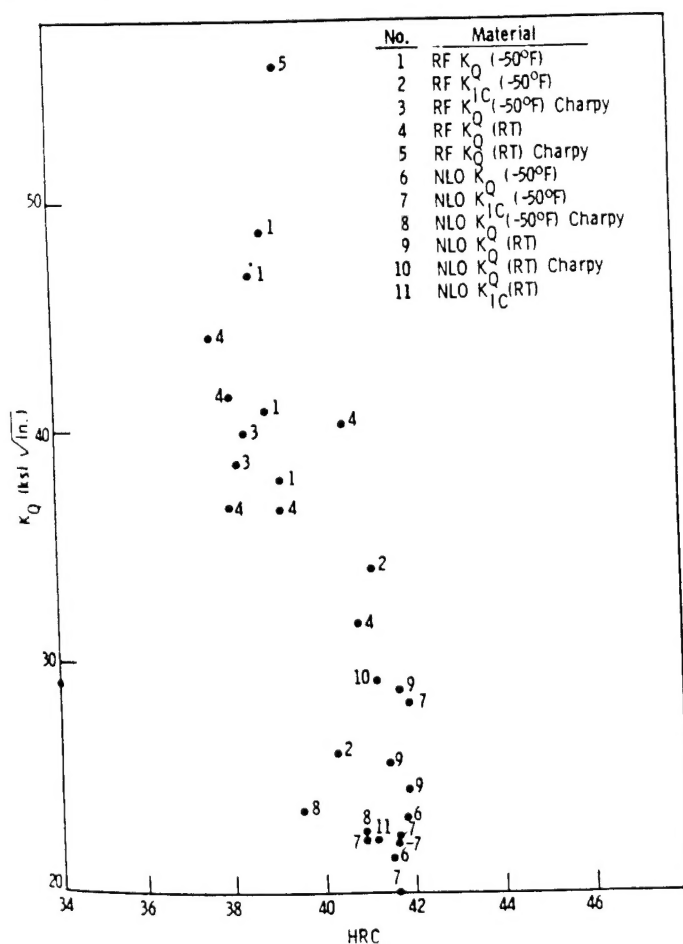


Figure 6. Fracture toughness of aged U-0.75 wt% Ti RF and NLO penetrators versus HRC.

As-Rolled NLO Bars Heat Treated at ARL-MD, Watertown

Chemistry, Mechanical Properties

The chemical composition of the as-rolled NLO bars is shown in Table 2. All chemical properties except hydrogen meet the requirements of the XM774 as-cast Staballoy core specification. The 1.8 ppm hydrogen exceeds the maximum requirement of 1 ppm. Table 3 summarizes mechanical properties for the alloy aged at three different temperatures: 350°C, 370°C, and 390°C. In all three cases, the mechanical properties meet or exceed the minimum requirements specified for the heat treated XM774 U-0.75 wt% Ti core alloy. Data from the unaged material is included for comparison.

Table 2. Chemical analysis data for as-received NLO bars

Ti 0.72% Top		C 14 ppm Top	
Ti 0.71% Bottom		C 23 ppm Bottom	
H 1.8 ppm Top			
Si	60 ppm	Mg	< 4 ppm
Fe	34 ppm	Ba	< 3 ppm
Al	14 ppm	Cr	2 ppm
Ni	10 ppm	Be	< 1 ppm
Pb	9 ppm	B	< 1 ppm
Mn	8 ppm	Sn	< 1 ppm
Cu	7 ppm	V	< 1 ppm
Zn	< 20 ppm		
Density = 18.64			

Table 3. Mechanical properties of aged U-0.75 wt% Ti NLO bars

	Hardness (HRC)	YS (0.2%)* (ksi)	TS (ksi)	Elong* (%)	RA* (%)
Unaged	36.2	93.8	187.4	17.9	16.5
Aged for 7 hours at					
350°C	37.5	108.0	192.0	17.2	17.4
370°C	39.0	109.1	196.0	13.9	18.7
390°C	41.5	115.6	206.2	12.5	14.9

All bars solution treated at 800°C for two hours, 850°C for one-half hour, and vertically water quenched at 21 in. per minute. *Average of 4 values

Fracture Toughness Versus Temperature

Fracture toughness (K_Q) of the above mentioned materials were determined utilizing precracked Charpy specimens at test temperatures

ranging from -100°F to 70°F. The data are recorded in Table 4 and plotted in Figure 7. It should be noted that a limited number of specimens were available for test. Generally, fracture toughness increased with test temperature. The unaged alloy (solution treated and quenched) gave the highest fracture toughness values. As the aging temperature increased, fracture toughness decreased. The bars aged at 390°C gave the lowest fracture toughness values. Fracture toughness (K_Q) values were greater than 35 $\text{ksi}\sqrt{\text{in.}}$ for all aged bars at the -50°F and higher test temperatures. These data show that the fracture toughness of the NLO material can be substantially improved by changing the heat treatment procedure from solutionizing in NUSAL and fully plunge quenching in oil to solutionizing in vacuum and vertically quenching in water

Table 4. Fracture toughness (K_Q) of aged U-0.75 wt% Ti NLO bars

	Test Temperature (°F)				
	-100	-50	-20	10*	R.T.*
	K_Q ($\text{ksi}\sqrt{\text{in.}}$)				
Unaged	33.8	38.35	46.4	54.0	61.55
Aged for 7 hours at					
350°C	34.6	36.30	41.3	48.2	54.95
370°C	29.5	35.20	43.9	43.1	58.45
390°C	28.8	35.60	39.2	42.7	43.2

All bars solution treated at 800°C for two hours and 850°C for one-half hour and vertically water quenched at 21 in. per minute

*Average of 2 values

Fracture Toughness Versus Hardness

Figure 8 plots fracture toughness (K_Q $\text{ksi}\sqrt{\text{in.}}$) versus HRC hardness for the unaged and aged bars. Room temperature fracture toughness values decreased significantly with increase in HRC hardness and aging temperature. At the -50°F and -100°F test temperatures the rate of decrease of fracture toughness values with increase in HRC hardness and aging temperature decreased markedly.

BNW Bars Vacuum Solution Treated, Vertically Water Quenched and Aged at 350°C for 16 Hours

Chemistry, Mechanical Properties

Table 5 shows that BNW processed alloy meets the chemical properties requirements of the XM774 specification. Note that the hydrogen content is 0.5 ppm.

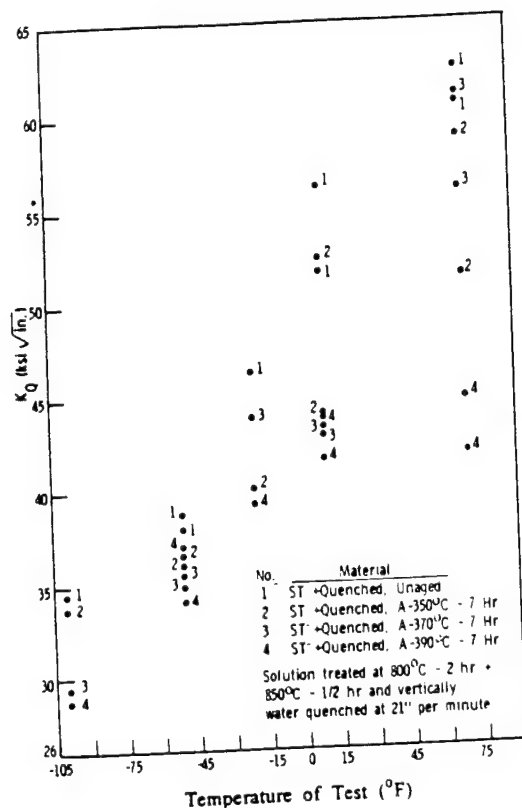


Figure 7. Fracture toughness of aged U-0.75 wt% Ti NLO bars versus temperature of test.

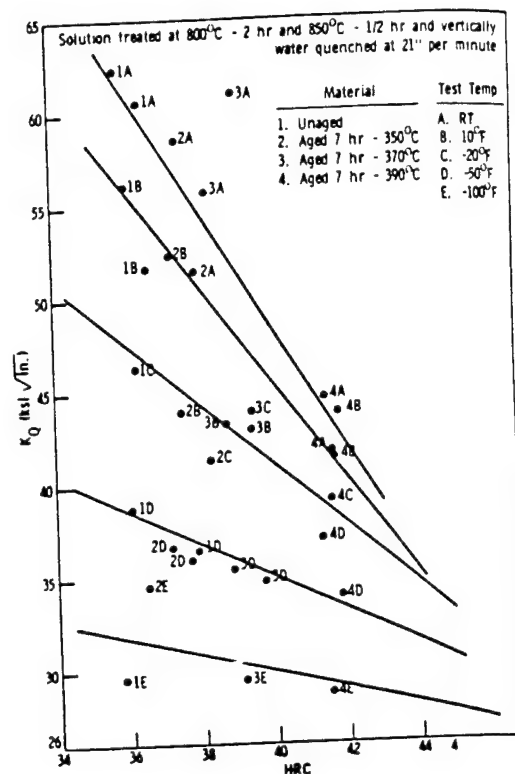


Figure 8. Fracture toughness of aged U-0.75 wt% Ti NLO bars (Charpy - K_Q) versus HRC

Table 5. Chemical analysis of BNW bars (101, 103, 104, 105, 107, 108) from 4-1/2 in. diameter ingot

Ingot Analysis			
Ti Center	0.73%	Si	45 ppm
Ti Bottom	0.73%	Fe	30 ppm
H	0.5 ppm	Nb	<10 ppr
C	70-80 ppm	Ni	25 ppm
Al	5 ppm		

Figure 9 summarizes HRC traverse data taken across the diameter of transverse sections for six bars at 45° angles at the vertically water quenched end, marked A (first hits H₂O), and 6 in. from the end, marked B. The bars at position B were slightly harder than at position A. The central areas of the bars were quite uniform in hardness and slightly softer.

The tensile properties of the six aged U-0.75 wt% Ti bars versus temperature are shown in Table 6. The yield strength (YS) was found to increase slightly with decrease in test temperature. The strength of the material exceeds the minimum requirements of the XM774 specification.

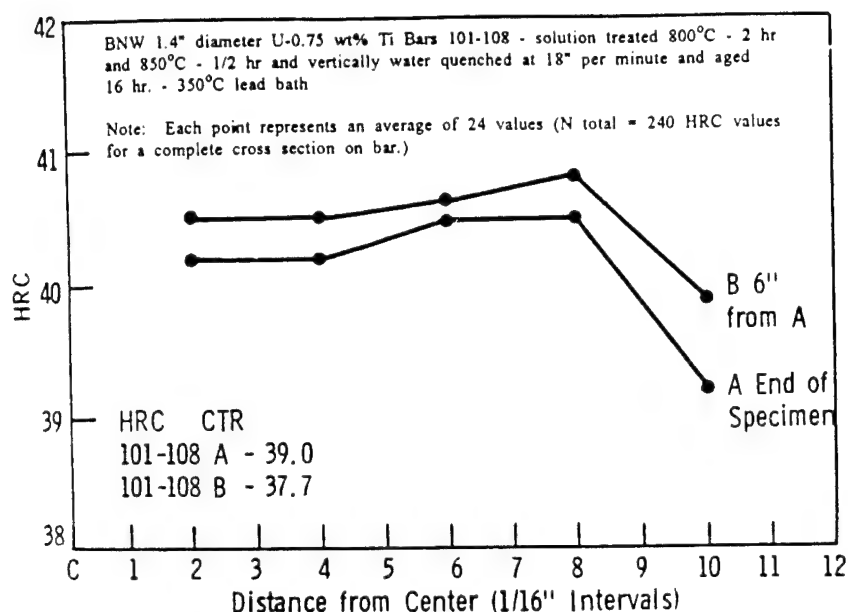


Figure 9. Transverse Rockwell C Hardness versus distance from center.

Table 6. Variation of tensile properties of aged U-0.75 wt% Ti BNW* bars with temperature

Temp (°F)	YS 0.1% (ksi)	YS 0.2% (ksi)	ULT (ksi)	E (psi x 10 ⁶)
70	101	114	199	20.5
40	104	116	196	19.3
10	102	115	206	18.5
-20	106	120	210	19.5
-50	110	124	206	19.5
-100	108	122	200	20.5

*Batelle Northwest 1.4 in. diameter U-0.75 wt% Ti bars #10 through 108 solution treated at 800°C for two hours, and 850°C for one-half hour, vertically water quenched at 18 inches per minute; aged for 16 hours at 350°C lead bath (4.5 in. diameter ingot α extruded).

NOTE: Average of 2 values

Fracture Toughness Versus Test Temperature

Figure 10 plots fracture toughness versus test temperature from -100°F to 100°F. Four test values were obtained at each temperature and lines were drawn through the outermost points to show the band of values. Fracture toughness increased with increasing test temperature. There was no evidence of change or decrease in slope at the +100°F test temperature, but below -50°F the slope decreased indicating a brittle region. The average K_{IQ} value for each test temperature is shown in Table 7. Note that the average K_{IQ} value at -50°F is 36.2 ksi $\sqrt{\text{in.}}$, which exceeds the recently established minimum XM774 requirement of 30 ksi $\sqrt{\text{in.}}$

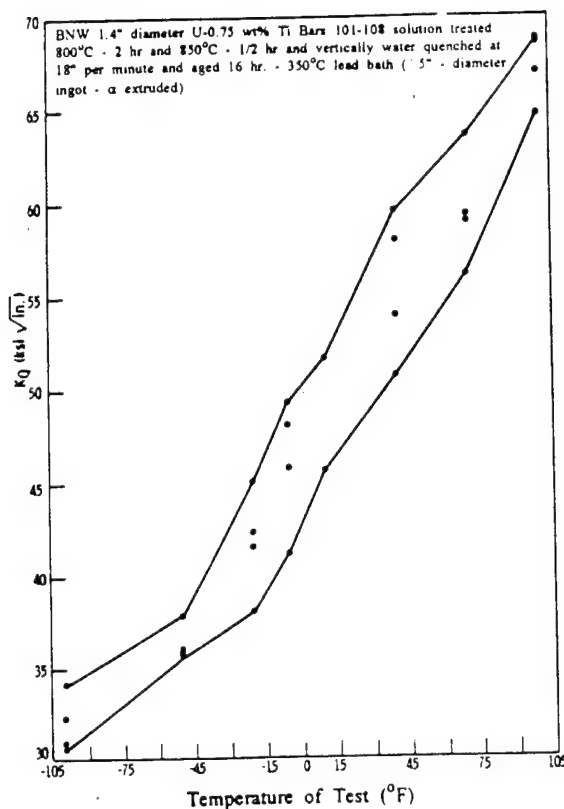


Figure 10. Fracture toughness of aged U-0.75 wt% Ti BNW bars versus temperature of test.

Table 7. Variation of fracture toughness of aged U-0.75 wt% Ti BNW bars with temperature

Temperature (°F)	Hardness* (HRC)	KQ† (ksi√in.)
100	39.4	67.2
70	39.5	59.4
10	39.7	55.6
-4	39.7	47.6
-20	39.6	46.0
-50	39.4	41.8
-100	39.4	36.2
	39.7	31.9

*Average of 16 values †Average of 4 values
Battelle Northwest 1.4 inch diameter U-0.75% Ti bars #101 through 108 solution treated at 800°C for two hours and 850°C for one-half hour; vertically water quenched at 18 in. per minute and aged 16 hours at 350°C in lead bath (4.5 in. in diameter ingot α extruded).

Stress Corrosion Cracking

Table 8 compares the critical stress intensity for crack propagation in an aqueous solution containing 50 ppm C1-(K_{ISCC}) for 1000 hours of the NLO processed XM774 U-0.75 wt% Ti alloy (solution treated in NUSAL and plunge quenched in oil and aged) with the RF processed

XM833 alloy (vacuum solution treated and vertically water quenched and aged). The RF XM833 U-0.75 wt% Ti alloy is less susceptible to stress corrosion than the NLO XM774 material due to the differences in processing. Crack extension in all of the alloys was transgranular and failure occurred by brittle quasicleavage fracture in NaCl solution (1,10).

Table 8. K_{Isc} data for 105 cal. penetrators in 50 ppm Cl-

NLO	XM774 (8 Specimens)	18 $\text{ksi}\sqrt{\text{in.}}$
RF	XM833 (6 Specimens)	23 $\text{ksi}\sqrt{\text{in.}}$

Ratio Analysis Diagrams (RAD)

K_{IC}/σ_{YS}

The best index of a material's fracture resistance is the K_{IC}/σ_{YS} ratio since it is this ratio of materials properties that determines flaw size and applied stress which are the parameters of interest to designers. The so-called ratio analysis diagram (RAD) (4,5) encompasses the range of strength and fracture resistance. Its framework is formed from the scales of YS versus K_Q . The technological limit line represents the highest values of fracture resistance measured to date.

Figure 11 contains the RAD constructed for the U-0.75 wt% Ti alloy (6-8). The envelope "B" encompasses fracture toughness data obtained for the NLO processed alloy which are representative of the failed (low temperature launch) penetrator lots. The material was molten salt solution treated, quenched in oil, and aged; it also contained high hydrogen (>1 ppm). Envelopes "A" and "D" contain data for penetrators which were vacuum solution treated, vertically water quenched, and aged with a low hydrogen content (<1 ppm).

The data shows that the fracture toughness of the alloy is highly sensitive to variations in heat treatment and concomitant interstitial content and microstructure. Under optimum conditions a fracture toughness of $80 \text{ ksi}\sqrt{\text{in.}}$ has been reported for the U-0.75 wt% Ti alloy at a YS of 115 ksi. Further processing improvements and alloy development may raise this current limit to $90 \text{ ksi}\sqrt{\text{in.}}$

K_{Isc}

The RAD shown in Figure 11 superimposes K_{Isc} data on the fracture toughness data displayed in Figure 12. The envelope shown contains earlier K_{Isc} data obtained in 50 ppm Cl- solution and

The graph plots $K_Q \text{ (Ksi)}\sqrt{\text{In}}$ on the y-axis (0 to 100) against Yield Strength (ksi) (0.2% Offset) on the x-axis (0 to 220). A solid curve represents the 'Current Technological Limit'. A horizontal line is drawn at $K_Q \approx 90$. Five regions are outlined: A (RF), B (NLO), C (GE - General Electric), D (NLO - HIT TMT (ARL-MD Watertown)), and E (LA). Region E is the largest, bounded by the technological limit curve and the horizontal line. Region D is to the left of E. Region A is a small region within D. Region B is a small region below A. Region C is a region to the right of B and below the technological limit curve.

Y-axis: $K_Q \text{ (Ksi)}\sqrt{\text{In}}$

X-axis: Yield Strength (ksi) (0.2% Offset)

Current Technological Limit

Legend:

- A - RF
- B - NLO
- C - GE (General Electric)
- D - NLO - HIT TMT (ARL-MD Watertown)
- E - LA

Figure 1 is a scatter plot showing the relationship between Yield Strength (0.2% Offset) on the x-axis and K_{IC} (ksi $\sqrt{\text{in.}}$) on the y-axis. The x-axis ranges from 75 to 200, and the y-axis ranges from 0 to 100. Five linear regression lines are shown, labeled 0.1 through 0.5, representing different K_{IC} / σ_{VS} ratios. Data points are plotted for various sources: G (G.E. GAU 8A Mantech), F (Frankford Arsenal), M (Magnani - ARL-MD), RF (Rocky Flats), and NLO (Naval Ordnance Laboratory). A cluster of points is circled and labeled "50 PPM Cl^- ".

Legend:

- G - G.E. GAU 8A Mantech
- F - Frankford Arsenal
- M - Magnani - ARL-MD
- RF - Rocky Flats
- NLO - Naval Ordnance Laboratory

Figure 12. SCC and K_Q RAD for U-0.75 wt% Ti alloys.

Cooperative Test Program with ARDEC

ARDEC provided ARL-MD, Watertown with additional U-0.75 wt% Ti alloy which was similarly processed by three suppliers: Nuclear Metals, Inc. (NMI), NLO, and BNW (all vacuum solutionized, vertically water quenched, and aged).

Fracture toughness measurements were made and the data reported in Table 9. At -50°F , values in the range of 31 to 41 $\text{ksi}\sqrt{\text{in.}}$ were obtained. Based on these data, it was recommended that a minimum fracture toughness requirement of 30 $\text{ksi}\sqrt{\text{in.}}$ at -50°F be incorporated in the XM774 core specification.

Table 9. Fracture toughness of U-0.75% Ti similarly processed by three suppliers

Lot No.	Temp. ($^{\circ}\text{F}$)	n	HRC	K _Q ($\text{ksi}\sqrt{\text{in.}}$)
		NMI		
48	69	2	42.4	46.4
	-50	4	41.5	31.4
72	69	2	39.2	59.4
	-50	4	38.9	37.3
		NLO		
732-734	74.8	4	41.9	52.8
	-50	8	41.4	34.9
		BNW		
*	73.8	12	40.4	66.3
	-50	23	40.2	40.9
307	-50	1	44.8	37.1
319	-50	2	43.3	36.3

*11, 83, 93, 152, 203, and 249 lot numbers

Conclusions

It was shown that the fracture toughness of the U-0.75 wt% Ti alloy is highly sensitive to variations in heat treatment and concomitant interstitial content and microstructure. The NLO processed U-0.75 wt% Ti alloy representative of the failed penetrators (low temperature launch) had appreciably lower fracture toughness ($\sim 20 \text{ ksi}\sqrt{\text{in.}}$ at -50°F) than the alloy processed either by BNW or RF ($\sim 35 \text{ ksi}\sqrt{\text{in.}}$ at -50°F).

The failed NLO material was characterized as high hydrogen content (2 to 4 ppm), low elongation (7%) material with microstructural features that included a coarse grain size, duplex structure, banding, and centerline porosity.

By comparison, the BNW and RF processed alloy contained less hydrogen (<1 ppm), exhibited higher elongation (14%), and essentially a martensitic structure with small voids in the central area. However, it was demonstrated that the NLO material could achieve comparability of fracture toughness to the BNW and RF processed alloy by solutionizing in vacuum, vertically water quenching, and aging instead of solutionizing in molten salt, fully plunge quenching in oil, and aging. Based on the extensive fracture toughness testing of XM774 core material similarly processed by several vendors for ARDEC, a minimum fracture toughness requirement of $30 \text{ ksi}\sqrt{\text{in.}}$ at -50°F should be incorporated into the XM774 specification to insure launch integrity.

The U-0.75 wt% Ti alloy is very susceptible to stress corrosion cracking in aqueous chloride solutions (K_{ISCC} 18 to $23 \text{ ksi}\sqrt{\text{in.}}$). Residual stress measurements of the fabricated M774 penetrator should be made to determine the magnitude of the tensile stress introduced by the processing of the alloy.

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